

BIODEGRADABLE GREASE TECHNOLOGY FOR FUTURE ARMY COMBAT SYSTEMS

In-Sik Rhee*

U.S. Army Tank-Automotive Research, Development, and Engineering Center
Warren, MI, 48397

ABSTRACT

Environmental safety and compliance has recently become the most significant worldwide issue. Over the past decades, many Army installations throughout the United States have been contaminated with petroleum hydrocarbon-based fuels and lubricants. The generation of the potentially hazardous wastes by Petroleum, Oil, and Lubricant (POL) products not only cause both short and long term liability with respect to environmental damage, but can result in deteriorated mission performance and high cleanup costs. For this reason, the Army is currently soliciting a technology that can be used to minimize waste stream of POL products utilized in the current and Future Combat Systems. This paper presents the results of development of Army's biodegradable greases, laboratory biodegradation tests, and field demonstrations including bioremediation tests.

1. INTRODUCTION

Soils and ground water at many military installations throughout the United States have been contaminated with petroleum and related fuels, lubricants and associated products, such as lubricating oils, greases, hydraulic fluids, engine fuels, and fuel oils [Klaus, 1993]. Currently, the Resource Conservation and Recovery Act (RCRA)[RCRA,1976] and the DoD Hazardous Waste Minimization (HAZMIN) Policy mandate that all DoD installations must reduce the quantity or volume and toxicity of hazardous waste generated by POL products wherever economically, practicable, and environmentally necessary. To achieve the HAZMIN goals, research and development efforts are being directed to develop new or improved HAZMIN techniques and processes.

In a survey, the Department of Defense (DoD) annually procures and uses a large amount of petroleum products and coolants in various applications. This results in a significant volume of used and off-specification products generated at DoD installations. To reduce this waste stream, recycling and refining technologies were recently introduced into the military community. Many lubricating oils are being recycled or re-refined, or can be burned for their energy value. Unlike oils, solid and/or semi-solid lubricants such as lubricating greases can not be considered for recycling due to difficulty in their liquefaction. Because of this, most military greases do not meet the DoD HAZMIN goals. For these reasons, many

military users have shown increased interests in the development of new, biodegradable greases which appears to be very promising for addressing this problem from the front end [H.F. Eichenberger, 1991, D. Sukys, 1994, N. Kato, 1999].

In response to the demand of military biodegradable lubricating greases (BLGs), a program was initiated to develop a multipurpose BLG product which can rapidly biodegrade and will be less toxic to environment while, at the same time, providing satisfactory field performance in future military combat systems. The purpose of this study was to replace the military automotive greases, MIL-PRF-10924G, Grease, Automotive and Artillery (GAA) [Military Specification, 1998] with BLG product. One of major impediment to the development of BLG products, is the lack of clearly defined criteria, which can reliably quantify the biodegradability and lubrication properties of a product. Because of this, the study has focused on the development of BLG products, biodegradability measurement and their lubrication properties.

2. DEVELOPMENT OF PRELIMINARY TARGET REQUIREMENTS FOR BLG

The Army has been using a National Lubricating Grease Institute (NLGI) Number 2 consistency grease covered by military specification, MIL-PRF-10924, as the standard grease for all military vehicles and artillery and ground equipment operated worldwide. This grease was originally designed for used in extreme field environments and multipurpose application. To meet these requirements, the formulation of the grease consisted of a blend of 6 cSt Polyalphaolefin (PAO) and high viscosity mineral oil thickened with an advanced lithium complex thickener system. This grease has a wide operational capability (-54 to 180 °C), excellent water and storage stability, good shear and oxidation stability, good antiwear and load carrying capacity, and saltwater corrosion properties. One disadvantage is its low biodegradability. For this reason, the MIL-PRF-10924G grease is not considered a biodegradable product.

To improve its environmental properties, new target requirements were developed based on the requirements of MIL-PRF-10924G specification and what is believed

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to be achievable with current biodegradable grease formulation technology. These target requirements are listed in Table 1. In these requirements, the American Society for Testing and Materials (ASTM) D5864 method was adopted for biodegradation tests with its readily-biodegradable criteria.

3. INTERIM PRODUCTS

Effort on the development of experimental biodegradable grease was directed toward comparing the target requirements of new proposed biodegradable MIL-PRF-10924H grease to the performance potential of available biodegradable materials and environmentally acceptable additive technology. To minimize a compatibility problem, a formulation guideline was developed based on a lithium complex technology with advanced additive formulation used in the MIL-PRF-10924G greases. Initially, three experimental BLG products were formulated with a grease manufacturer. First, two experimental greases, BLG (1, 2) were reformulated based on non-biodegradable MIL-PRF-10924G grease formulation, while BLG (3) was formulated with a low viscosity blended ester oils. The MIL-PRF-10924G grease was originally formulated with 65% by weight of polyalphaolefin (PAO) oil, 10% of petroleum oil, 15% of lithium-complex thickener, and 10% of the additives. In an earlier study, it was found that this formulation has a low biodegradability due to the non-biodegradable base oils. To improve its biodegradability, this formulation was modified using a blend technique that is often used with success in the formulation of the conventional petroleum-based greases. A rapeseed oil (vegetable oil) was selected as a biodegradability improver based on its high biodegradation performance. Initially, an experimental BLG (1) was prepared adding 10 percent of rapeseed oil into MIL-PRF-10924G, while BLG (2) were reformulated by adding 20 percent of rapeseed oil while deleting the petroleum oil and some PAO oil from this formulation. In another formulation, BLG (3) was a fresh formulation with a blend of polyol ester, diester and PAO. Table 2 provides the composition of BLG (1), BLG (2), and BLG (3) as compared to the baseline MIL-PRF-10924G grease.

4. BIODEGRADATION TEST

Biodegradation is a natural process caused by the action of microorganisms. In the presence of oxygen, nitrogen, phosphorous, and trace minerals, organic pollutants can support microbial growth and are converted into a series of oxidation products that generally conclude with carbon dioxide and water. The biodegradation test method adopted in this study follows the ASTM D5864,

Table. 2 Experimental Biodegradable Lubricating Greases

Composition	MIL-PRF-10924G	BLG (1)	BLG (2)	BLG (3)
Base Oil	PAO +Mineral	PAO +Mineral	PAO	Polyol ester +Diester + PAO
Biodegradation Improver, (Rapeseed Oil)	None	10 %	20 %	None
Li-Complex Thickener	15	15	15	15
Other Additives	10	10	10	10

* BLG - Biodegradable Lubricating Grease

“Standard Test Method for Determining Aerobic Biodegradation of Lubricants and Their Components”. Recently, ASTM D-12 Subcommittee on Environmental Standard of Lubricants has developed this biodegradation test method based on the OECD Modified Sturt Test which closely simulates the wastewater biotreatment conditions [Donna, 1995]. This test method was originally designed to determine the degree of aerobic aquatic biodegradation of lubricants on exposure to an inoculum under laboratory conditions. In this test, the biodegradability of a lubricant is expressed as the percentage of maximum (theoretical) carbon conversion (or carbon dioxide generation) under well-controlled conditions for a period of 28 days.

To evaluate the biodegradability of interim products, a biodegradation test was conducted according to the ASTM D5864 test method. A commercially available rapeseed based lithium grease and MIL-PRF-10924G grease were also tested as the baseline of this study.

Test Apparatus: A schematic diagram of the biodegradation test apparatus for ASTM D5864 test is shown in Figure 1. This test apparatus was a slightly modified for the study. It consists of four separate units: the air supply/carbon-dioxide scrubbing system, the incubation/biodegradation batch reactor, a carbon-dioxide collector, and a titrator. Both the carbon-dioxide scrubbing and the biodegradation units utilize Erlenmeyer flasks. To eliminate other carbon sources, except the test lubricant, CO₂-free air is needed for the biodegradation test. A laboratory compressed air supply was attached directly to the carbon dioxide scrubbing system to produce CO₂-free air. The scrubbing system uses cascade

flasks: two flasks containing 10 M potassium hydroxide (KOH) solution and two flasks containing 0.025 M barium hydroxide $\text{Ba}(\text{OH})_2$ solution. To ensure the desired aerobic environment, the test solution containing the test lubricant was fully agitated using a variable speed magnetic stirrer. In order to conduct multiple tests for performance comparison, ten separated identical biodegradation batch reactors were connected as seen in Figure 1. Each reactor can be independently operated for aquatic biodegradation tests.

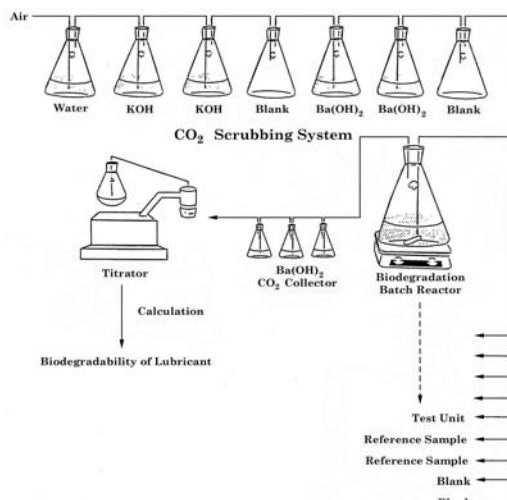


Figure 1. Schematic Diagram of Biodegradation Test Apparatus

Test Procedure: Prior to the test, the test solutions were prepared according to the road diagram shown in Figure 2. Initially, five stock solutions for the test medium were prepared: ammonium sulfate solution (40 g/L), calcium chloride solution (27.5 g/L), ferric chloride solution (0.25 g/L), magnesium sulfate solution (22.5 g/L), and phosphate buffer (made of 8.5 g potassium dihydrogen, 21.7 g potassium monohydrogen phosphate, 33.4 g sodium monohydrogen phosphate, and 1.7 g ammonium chloride). It should be noted that these solutions do not contain any carbon material in order to avoid an extra source of carbon dioxide production. For a positive control and a fair comparison, a rapeseed oil was used as a reference sample. This oil has been identified to be a high biodegradable material. To determine the theoretical CO_2 evolution, the initial carbon content of the test lubricants was analyzed using the LECO CHNS-932 Elemental Tester [LECO, 1992].

The sewage inoculum (i.e., bacteria, yeast, fungi) was carefully prepared from the mixed liquor (approximately 1 liter) of activated sludge provided by a local wastewater treatment plant. This sewage inoculum was freshly collected from the biotreatment processing pool of the

plant. It was fresh and contained the proper microorganisms for treating regular wastewater. In our laboratory, the total number of bacteria and fungi were observed and counted using the Easicult Bacteria Counting Kit. This test kit is commonly used for measuring the growth of bacteria in an industrial process, such as cutting fluids, etc. To avoid carry-over of sludge solids which might interfere with the measurement of CO_2 production, the sewage inoculum was homogenized by a blender and aerated until ready for use.

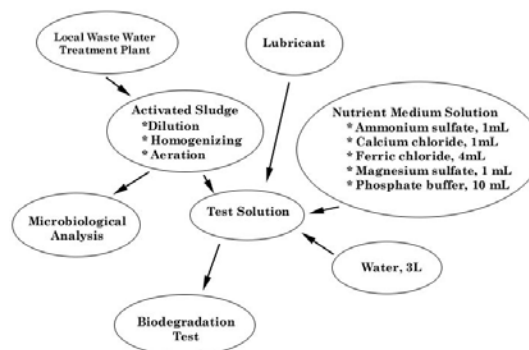


Figure 2. Preparation of Test Solution

A total of ten test flasks were used for the biodegradation tests. Six flasks were designed for the test samples, two flasks as positive controls (baseline reference sample: rapeseed oil), and the remaining two flasks as blank controls. To prepare a one-percent inoculum solution, 2,470 mL of distilled water was added to each 4 L Erlenmeyer flask. Then, the following stock solutions (concentration explained earlier) were added to the test flasks: 3 mL of ammonium sulfate, magnesium sulfate, and calcium chloride; 30 mL of phosphate buffer; and 12 mL of ferric chloride stock solution. After all stock solutions were mixed and diluted in the 4 L Erlenmeyer flasks, 30 mL of the activated sludge inoculum mentioned earlier was added to the test solution.

To purge the CO_2 gas that might have migrated from the room air of the laboratory during the inoculum solution preparation period, the test flasks were aerated with CO_2 -free air for 24 hours. Then, three CO_2 collectors filled with 100 mL of 0.0125 M $\text{Ba}(\text{OH})_2$ solution were connected in series to the downstream of each 4 L Erlenmeyer flask. Before adding the test (or reference) lubricant samples, the pH values of the test solutions were all adjusted to 6.5 - 7.5 using HCl or NaOH solutions. About 80 mg of the test lubricant sample was added into each of the six test flasks. Rapeseed oil was also added into the duplicate positive control flasks. It is noted that the duplicate blank control flasks were free of test lubricants. Then, the distilled water was added to maintain a final volume of 3 liters in each 4 L flask. All

test flasks were tightly stoppered and maintained at 20 - 25 °C. The magnetic stirrer was kept at approximately 200 rpm. The volumetric flow rate of CO₂-free air to each test flask was maintained at 50 to 100 mL/min. It should be noted that each experiment included duplicate control flasks and duplicates of each test lubricant sample. During the test, the test room was kept in complete darkness. This measure was necessary to prevent photo degradation of the test substance and the growth of photosynthesis bacteria and algae.

To measure carbon dioxide production during a predetermined test period, the CO₂ collector nearest the 4 L Erlenmeyer flask was removed for titration and calculations. The remaining two collectors were moved up one place closer to the 4 L Erlenmeyer flask and a new collector filled with 100 mL of fresh 0.0125 M Ba(OH)₂ was placed at the far end of the series. Titration was performed every day for the first 10 days and then every other day for the remaining 18 days or until a plateau of CO₂ evolution was reached. The end point used for automatic titration was set at pH 7. Once the CO₂ evolution has reached a plateau, the pH of the test solutions were measured and added 1 mL of HCL into the test solutions to decompose the inorganic carbonate and to release trapped CO₂ for a final titration. Data obtained from the titration were converted to the amount of CO₂ production using an equation specified by the method.

Test Results: A summary of measured biodegradation results of five test samples is plotted in Figures 3. It is noted that these results were determined based on duplicate test results in order to increase reliability of test data and are expressed as percentage of the maximum (theoretical) carbon dioxide evolution of each test sample. Figure 3 shows that the biodegradability of 28 days ranges from 28% for MIL-PRF-10924G grease to 76.8% for Rapeseed based grease. The BLG (3) provides a biodegradability of 64 %, while experimental BLG (1) and BLG (2) mark 46.5% and 45.3%, respectively. All BLG products exhibited a higher biodegradability than MIL-PRF-10924G grease. It may be stated that greases from either natural source or synthetic ester derives are better biodegradable materials than petroleum-based products. Especially, the BLG (1) with 10% rapeseed oil gave 66 % improvement of biodegradability over the baseline grease, while BLG (2) with 20 % rapeseed oil improved the biodegradability by almost 59 %. Even though both results gave a lower value than the biodegradability of 100% rapeseed-BLG, it may generally be concluded that a small amount of biodegradability improver (rapeseed oil) which bacteria like to eat due to its natural ester flavor may substantially improve the biodegradation process of a lubricant. Also, Figure 3 demonstrates that the biodegradation process of rapeseed-BLG starts earlier than the other three greases and slows after first week of test. The BLG (2) with 20% rapeseed

oil provides a similar biodegradation profile to rapeseed-BLG, while the biodegradation behavior of BLG (1) with 10% rapeseed oil was similar to its base grease (MIL-PRF-10924G). Unlike the other types of greases, the synthetic ester based BLG (3) tends to steadily increase the biodegradability over 28 days. It appears that the biodegradability of greases highly depends on the types of base oils used in the grease formulation.

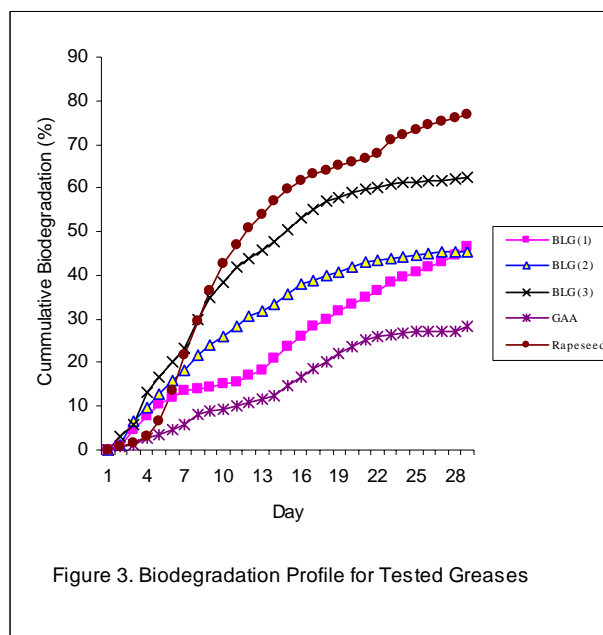


Figure 3. Biodegradation Profile for Tested Greases

5. PERFORMANCE CHARACTERSTICS OF BLG

Military greases are currently used in various mechanical equipment designed for open lubrication system such as automobile wheel bearings, chassis systems, and gearboxes. As a replacement of current GAA grease, the experimental BLG products were also designed for use in the same mechanical applications. To evaluate the performance of experimental biodegradable lubricating grease, the BLG (3) was only selected based on its high biodegradability among the interim products. For comparison purposes, a product qualified under MIL-PRF-10924G and Rapeseed-BLG were also tested for the baseline study. These greases were evaluated according to the test protocol and the results are summarized in Table 1.

The most distinguishing property of grease is its consistency that is related to the hardness or softness of the grease. The consistency is rated by the penetration number. It is defined as the depth, in tenths of a millimeter, that a standard cone penetrates a sample of

grease under prescribed conditions of weight, time and temperature. To ensure a uniform sample, grease is worked 60 strokes using a grease worker before running the penetration test. The results are classified by grade ranging from 000 (very soft) to 6 (very hard) using National Lubricating Grease Institute (NLGI) grease classification system [NLGI, 2004]. The test results showed that all samples except for Rapeseed-BLG rated as NLGI No. 2 grade which marked as medium consistency. It indicated that the vegetable oils can also be used as base oil of greases like petroleum-based oils.

The mechanical stability of greases is usually evaluated by the work and water stability tests. In these tests, the stability of greases is determined based on the measurement of penetration changes in consistency due to the continuous application of shearing forces with and without water present. If grease has a mechanical stability problem, it will usually appear normal before being subjected to service but will soften rapidly or harden upon working. Concurrently, it can lead to a lubrication failure in mechanical components. In these tests, Rapeseed-BLG had substantial water problem in stability, while BLG product did not show any abnormal behavior. It appeared that vegetable-based BLG products have some degree of mechanical stability problems under wet condition.

The thermal stability of the lubricating greases is comprehensively evaluated using the results obtained from the dropping point and evaporation tests. The dropping point tends to measure the high temperature operability of greases and dependent on the type of thickener used in grease. A high dropping point grease usually provides better thermal stability for extension of bearing life at high temperatures. Rapeseed-BLG showed a medium dropping point, while both BLG (3) and MIL-PRF-10924G grease had a high dropping point due to their lithium-complex thickener system. The evaporation loss at elevated temperatures also indicates the degree of the thermal stability of greases. Rapeseed-BLG had a higher evaporation loss than the other two greases due to its base oil. The BLG (3) product provides excellent thermal stability at this test temperature (180 °C).

Oxidation stability is another important property of biodegradable greases and is intended to predict their storage and service life. To evaluate oxidative life of biodegradable lubricating greases, oxidation tests were conducted using ASTM D5483, Pressure Differential Scanning Calorimeter (PDSC) method. This method was developed to assess oxidation stability of the lubricating greases by measuring the differential heat flow between sample and a reference thermocouple at various temperatures (155, 180, and 210 °C) under a pressure of 3.5 MPa. In this procedure, the degree of oxidation stability at a given temperature is determined by

measurement of induction time. The PDSC test results showed that Rapeseed-BLG had a lower induction time than those of the other two greases. This result also agreed with the results of the thermal stability tests.

Tribology (friction, wear, and lubrication) properties are one of the important operational parameters in conventional mechanical systems. Most lubricating greases often use anti-wear additives to improve their wear prevention properties. This property is usually evaluated by the ASTM D 2264 Four-Ball Wear Test. The test results indicated that biodegradable lubricating greases do not have any compatibility problem with conventional anti-wear additives. This result was confirmed by ASTM D 2596 Four Ball Extreme Pressure (EP) tests.

Excessive oil separation of greases often indicates grease degradation in service/storage period. To assess this physical property, a static oil separation test was conducted using the Federal Test Method 791.321[FTM, 1986]. The results did not indicate any abnormal oil separation from either biodegradable lubricating greases. To verify this result, dynamic oil separation tests were conducted using the modified ASTM D4534 Method, Oil Separation from Lubricating Grease by Centrifuging (Koppers Method). The results showed that both biodegradable lubricating greases were physically stable under centrifugal force and provided the same quality of performance observed with the MIL-G-10924F grease.

For corrosion protection property, both biodegradable lubricating greases did not exhibit any corrosion problem with copper metal. In the saltwater rust tests, Rapeseed-BLG gave medium corrosion spots on the test bearing surface, while the other two greases did not show any corrosion problem. This chemical property is an important operational parameter for military readiness.

The low temperature property of greases is another important operational parameter in mechanical systems. If grease becomes too hard under sub zero temperatures, mechanical systems such as bearings, can lose lubrication and require higher torque. This can result in fatigue failure of mechanical system. Currently, this property is measured using a mechanical torque tester that was simulated from an automobile wheel bearing system. For the evaluation, three grease samples were tested at - 54 °C, using the US Army Low Temperature Torque test procedure [In-Sik, 1989]. The results showed that vegetable-based grease have very limited low temperature capabilities, while synthetic-based grease provides excellent rheological properties. It appeared that vegetable-based grease (Rapeseed-BLG) contains a wide range of fatty acid esters and tends to crystallize into long-chained esters of fatty acids well above the measured pour point.

The high temperature service life of grease is an another important operational parameters in mechanical systems. This property usually defines the upper operational temperature in service. Currently, several functional test methods are available to measure grease high temperature life. Among them, ASTM D3537 Method, Life Performance Test of Lubricating Greases, is widely used in the grease industry and by users. This method comprehensively evaluates all individual physical properties of greases directly related to high temperature and shear, using a simulated front wheel bearing system and a dynamic laboratory bench-type test apparatus [In-Sik, 1987]. Table 3 showed that the Rapeseed-BLG provides only 30 hours of high temperature life due to its poor thermal and oxidation stability. Such a grease can not be used in high temperature applications such as wheel bearings. On the other hand, the experimental BLG (3) product provided excellent high temperature life and its performance is equivalent to that of MIL-PRF-10924G grease.

6. FIELD DEMONSTRATION

To verify the functional performance of experimental BLG (3) product, field demonstration was initiated at Fort Hood and Fort Bliss, TX. A total of 10 military vehicles and equipment were utilized for this demonstration. The duration of this field demonstration was encompass a year testing period. Per the test plan, Fort Hood evaluated a BLG (3) using four military vehicles (i.e., HMMWV, 5-ton trucks), while Fort Bliss evaluated the experimental grease using five vehicles (i.e., contact maintenance truck, cargo truck, dump trucks) and a construction equipment under desert environment [In-Sik, 2001].

For the functional test, a BLG (3) was lubricated at Constant Velocity (C.V) Joints of HMMWV and front wheel bearings of other selected vehicles. These vehicles were used daily for their routine operation and military exercise. The evaluation criteria used in this demonstration were their field performance (i.e., consistency, oxidation, wear and corrosion problem, oil separation, etc.) and environmental performance (i.e., biodegradability, toxicity). Quarterly, the candidate grease and parts (i.e., C.V. Joints, bearings) of the tested vehicles were inspected and the grease samples were collected from each vehicle for the laboratory chemical analysis (i.e., PDSC, TGA). We also recorded operated mileage, environmental temperatures, and road conditions. In this demonstration, the operated mileage from the test vehicles ranged from 343 miles to 5,446 miles. These mileages were normal peacetime military usage for a year. As a result, a candidate BLG (3) product did not



Figure 4. BLG Field Demonstration at Fort Hood

give any abnormal behavior and clearly demonstrated the acceptable performance equivalent to the current military grease (MIL-PRF-10924G). In addition, it did not show any biodegradation in the tested bearing systems during the field demonstration period. This indicates that BLG product can only be biodegraded under certain environmental conditions (i.e., ground soil or water). The laboratory test results obtained from field samples also clearly supported these test results.

To verify the laboratory biodegradation results, a field biodegradation test was also set up at the bioremediation site of Fort Hood, TX. A candidate BLG (3) is being tested using a modified Fort Hood's remediation procedure. For reference purpose, the conventional MIL-PRF-10924G grease and Rapeseed-BLG were also tested at Fort Hood. For the test, the samples were mixed with soil and then, plowed and tilled in order to increase homogeneity. No fertilizer and microbes were applied except for water. Soil samples were obtained on a monthly basis and analyzed for total carbon content using the EPA method 413.2, Spectrophotometric, Infrared for Total Hydrocarbon from Wastes. The test results have clearly shown the rapid biodegradability of BLG (3) and Rapeseed-BLG as compared to the extremely slow biodegradability of conventional MIL-PRF-10924G grease. Both BLG products have degraded within five months. The data obtained from the field biodegradation test were plotted in Figure 4 and demonstrated a good correlation to the laboratory biodegradation test used in this study.

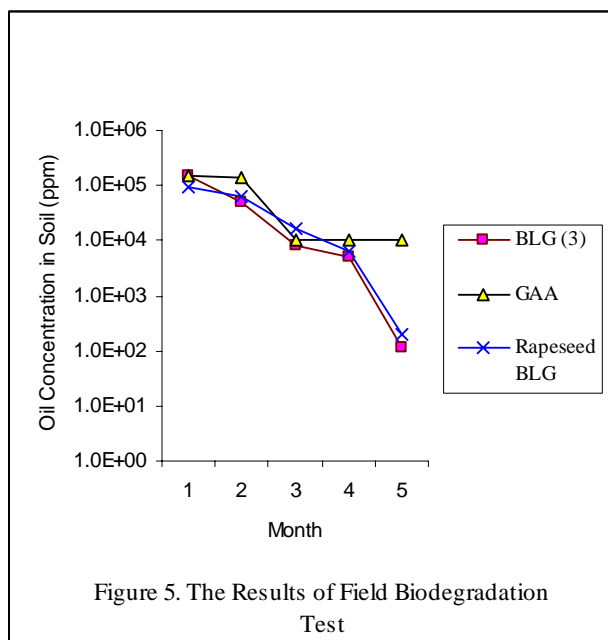


Figure 5. The Results of Field Biodegradation Test

7. CONCLUSIONS

Biodegradable greases have been comprehensively studied in order to evaluate their biodegradability and lubrication performances against the conventional petroleum based-greases. Synthetic ester based BLG (3) formulated for this study provided excellent biodegradability and exceptional lubrication performance under extreme conditions. This grease was specially blended with three different types of biodegradable oils (i.e., polyol ester, diester, polyalphaolefin) in order to meet the requirements of a proposed military biodegradable grease specification. The test results showed that BLG (3) product was compatible with conventional non-biodegradable synthetic oil-based grease and met all requirements of the designated specification that was designed for extreme environments. In addition, the overall performance of this BLG product has been proven in the field demonstration and accepted by military users. Therefore, the BLG (3) product can be used as the next generation of Army grease and will meet the requirements of future Army combat systems.

Biodegradable greases also require additive to enhance performance. The lubrication properties of some synthetic esters-based greases can be improved by antioxidants, corrosion inhibitors and wear/EP additives. However, the use of conventional additives in biodegradable greases may pose potential problems on their biodegradability. Especially, rapeseed oil used as a biodegradability improver to a conventional lubricating grease can distinctly improve its biodegradability.

Vegetable- and synthetic ester-based greases were found to exhibit excellent biodegradability over the conventional mineral-based products. However, rapeseed-based grease provides a limited operational performance due to the poor thermal and low temperature stabilities. On the other hand, lithium-complex greases formulated with synthetic ester gave better biodegradability than the PAO based-grease.

Experimental biodegradation test apparatus, devised based on the ASTM D5864 test method, was acceptable to evaluate biodegradability of greases and can be used in the grease quality control. Also, the test results were found to be a good agreement with field biodegradation performances.

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REFERENCES

- Donna Hoel, "Lubricants and Environments", ASTM Standardization News, June 1995.
- D.Sukys and C.Camino, "Natural Ester Biodegradable Fluids and Lubrication Trends for the Future", J of National Lubricating Grease Institute, Vol. 58, No.6, pp. 23-25, 1994.
- Federal Test Method Standard No. 791C, Lubricants, Liquid Fuels, and Related Products; Methods of Testing, 1986.
- H.F. Eichenberger, "Biodegradable Hydraulic Lubricants and Overview of Current Developments in Central Europe," 42nd Annual SAE Earthmoving Industry Conference, SAE Paper Series No. 910962, 1991.
- In-Sik Rhee, "Development of A New Test Method for Assessing Military Grease Performance at Low Temperatures", J of National Lubricating Grease Institute, Vol. 53, 1989.
- In-Sik Rhee, Developing Accelerated Endurance Test for Grease-A Status Report", US Army BRDEC Report, No. 2453, August, 1987.
- In-Sik Rhee, "Field Demonstration for Biodegradable Military Multipurpose Grease", U.S. Army TARDEC Report no. TR-13804, 2001.
- Klaus Pelinka, "Environmental Restoration Effort Takes on Momentum", National Defense, 1993.
- LECO Technical Manual, LECO Instrument Company, 1992.
- Military Specification MIL-PRF-10924G, Grease Automotive and Artillery, 1998.
- N. Kato and H. Komiya, "Lubrication Life of

Biodegradable Greases with Rapeseed Oil Base”, J. of the Society of Tribologists and Lubrication Engineers, pp.19-25, 1999.

NLGI Lubricating Grease Guide, pp. 3.4, NLGI Publication, 2004.

The Resource Conservation and Recovery Act of 1976,

42 U.S.C. 6901 et seq., 1976; the Clean Water Act of 1972 and amended in 1987.

Table 1. Laboratory Performance Test Results for Biodegradable Greases

Test Method		Target Requirement	Rapeseed-BLG	BLG (3)	MIL-PRF-10924G (GAA)
Dropping point, °C	ASTM D2265	240, min	178	272	278
Worked penetration, 1/10 mm	ASTM D217	265-295	250	289	282
Work stability, 100,000 strokes	ASTM D217	-25 to 60	+61	+32	+18
Roll stability	ASTM D1831	-25 to 60	+22	+15	+9
Evaporation loss, %, 180 °C, 1 hr	TGA	15, max	2.67	12.1	25.0
Oil separation, %	FED.791.321	10, max	2.5	2.79	1.6
Centrifuge, oil separation, 2hrs, 40 °C, %	Mod, ASTM D4425	18, max	3.8	17.2	8.8
Four Ball EP, Load Wear Index (LWI), kgf	ASTM D2596	30, min	27	38.5	42.2
Four ball wear scar dia., mm	ASTM D2264	0.6, max	0.58	0.53	0.48
Copper corrosion	ASTM D4048	1b	1b	1a	1b
Water stability, 1/10 mm	Mod. ASTM D217	-25 to 60	+100	-8	+41
Saltwater corrosion, 1% NaCl	Mod, ASTM D1743	No corrosion	Medium corrosion	No corrosion	No corrosion
Low temperature torque, -54 °C, N·m	Army method	Breakaway: 7 Running @5min:5, max	31.6(B) 4.63(R)	3.1(B) 1.5(R)	4.2(B) 2.2(R)
PDSC, min	ASTM D5483	10, min	14.3 (155 C)	23.6 (210 C)	12.9(210 C)
Biodegradability, %	ASTM D5864	60, min	48.9	64	28
Grease life, min, hr	ASTM D3527	100, min	30	100	130